

Fieldable Microsystems II (FMII)

David Fries and R.T. Short
University of South Florida
Marine Science Department
140 7TH Avenue South
St. Petersburg, FL 33701

Phone (727)553-3961 Fax (727) 553-3967 Email: dfries@marine.usf.edu
Phone (727)553-3961 Fax (727) 553-3967 Email: tshort@marine.usf.edu

Grant #: N00014-03-1-0480

LONG-TERM GOALS

The overall goal of this work involves the development of a self sufficient, fieldable micro sensor fabricated with micromachining technology. The targeted integrated sensor comprises both a chemical sensor (e.g. explosive detection) and physical sensor (CTD).

OBJECTIVES

The main focus of the effort is to advance an adaptive chemical analyzer that utilizes on-chip reaction separation and either a photonic or an electrochemical detection strategy. The scope of work included multiple developments: field deploy a separations device; advance seawater sampling preconcentration, devise an integrated opto chip; develop miniature HPLC that includes a mini high-pressure pump and mini power supply; place temperature and conductivity and depth functions on a planar surface and develop sensor packaging techniques.

APPROACH

As in the past we rely on the advances in our PCBMEMS fabrication methods that we have developed for ease of creation of the MEMS and to further the microsensors for the field. The chemical separation sensor has two main paths of development: ship board CE system and mini HPLC field sensor. Method development for seawater matrices is a necessary task for all field analysis. The current activity includes the transfer of the lab based chemical processor for deployment in the field along with advancing the CTD physical sensor.

WORK COMPLETED

Separations type micro-Chemical Sensor Field Unit

- Further the CE unit for the field
- Further the seawater sample pretreatment process and detector integration.
- Advance towards the integrated microchip HPLC unit
- Advance the ejection of chemicals off of the microchip for other detectors

Robotic Electronic Nose/Tongue

- Further the CE unit for amino acid fast detection

- Flume Demo a Carbon Fiber TNT sensor

Integrated Physical Sensing Device

- Continue on with the conductivity and temperature fabrication and test
- Implement a pressure function on chip

Sensor Array Development

- Advance the progress of the MEMS physical sensor package

Fabricate a group of sensors and test locally

Shipboard CE system. We have a completed lab system and have resolved the purification route for shipboard deployment for protein distribution measurements. We have scheduled deployment of the protein chip analyzer in the November 2003 in Florida Bay.

HPLC- pump. For the HPLC system we have focused effort on the HPLC column fabrication beyond the pressure pump work previously reported. We have advanced methods for creating monolithic HPLC stationary phases and microextraction (titania-PDMS) phases using sol gel media. The media is the preferred stationary phase in the HPLC chip and is the basis for a sample prep patent. We have created microchannels in liquid crystal polymer and are further integrating solid phase media with the channel architectures.

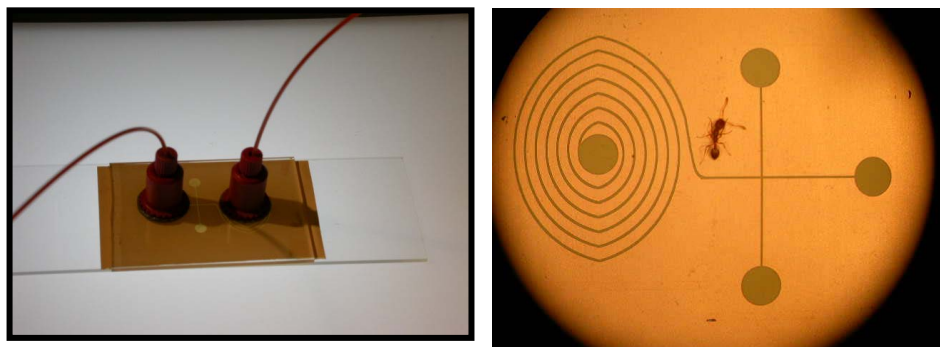


Figure 1. HPLC chip in liquid crystal polymer PCBMEMS. Fluidic interconnected system (left) and microchannel layout (right).

Micro Power Generation. We have made the components for a Cu/Zn Daniell's microbattery using our PCBMEMS technology. We have reported on the battery results to 1st International Microchannel Conference and also at a Fuel Cell Conference. We are primarily focusing on the PCBMEMS battery technology at this time and from this research are inserting an aluminum based PCBMEMS battery in BSOP (ONR funded drifter platform).

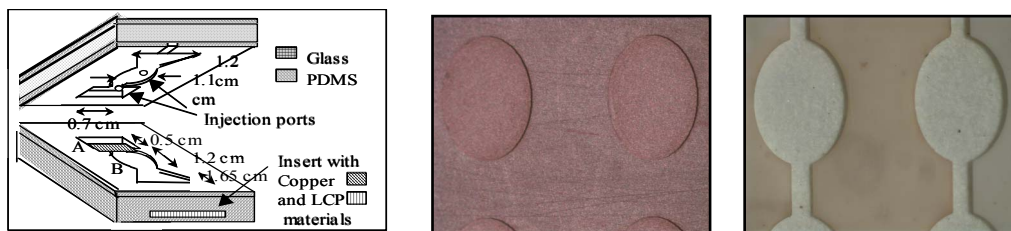


Figure 2. Isometric view of the micro-fluidic cell for Cu-Zn battery. Cu cathode (center) and electrolyte reservoirs (right) in LCP.

Opto-electronic cube. The compact LED/PD based opto-analyzer has made progress. We have designed, fabricated and are testing a disposable cube analyzer for explosives residues and other environmental targets using embedded solid phase extraction. Our approach is to use these detectors coupled tightly with AUV's for a robotic chemical screening. We are concluded the period with further testing of the sensor on the EEG Concepts ROV. Field data is shown for Cu detection from a field trial.



Figure 3. Robotic Chem Sensor testor (left). Remotely guided vehicle for testor field trails (center). Results from Cu sensitive solid phase material on RGV.

Electrochemical Detection system/nose. We have made continued progress for the electrochemical detection mode of the amino acid CE device and pressure driven devices. with a focus on explosives detection. In order to make a fieldable system we have expedited the deployment of the three-electrode voltammetric remote sensor. The probe has proven results for seawater analysis for explosives. We have made a submersible system and have made a recent field deployment using the canister with exceeded our target of a simple flume demo as part of this period of performance

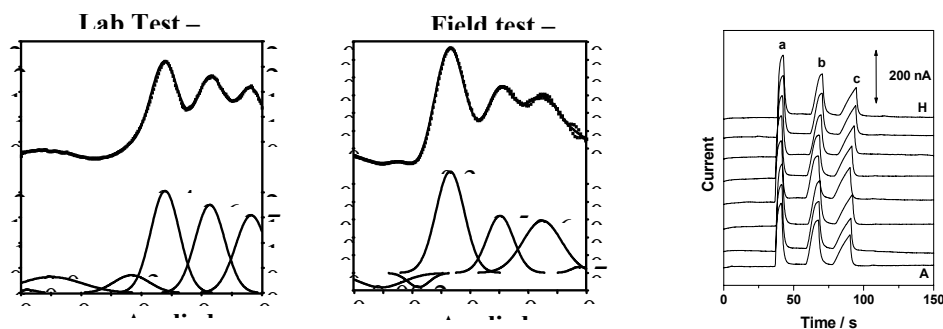


Figure 4. Square-wave Voltammograms for field test results in Tampa Bay, compared to laboratory experimental result. Explosive residues data indicating difference in signal from seawater and in proximity of TNT concentrate dispenser from sensor system in field deployed on RGV. Right- fast amino acid amperometric detection on chip using Cu electrode as an biomimetic detector.

MicroCTD. We have progressed the C, and D portion of the CTD. The thin film temperature is complete. We have focused much of our effort on liquid crystal polymer (LCP) for creating this PCBMEMS system due to its superior water resistance and the process steps for processing the LCP material. We are processing two C-cell modes at this point: phase measurement and electrode based. The phase technique measures the phase difference between two equal frequency RF signals coupling through the seawater medium through the two coils.

Initial tests yield a linear slope for change in temp at a fixed salinity. An integrated RF board with integrated RF devices is being fabricated into a single small PCB.

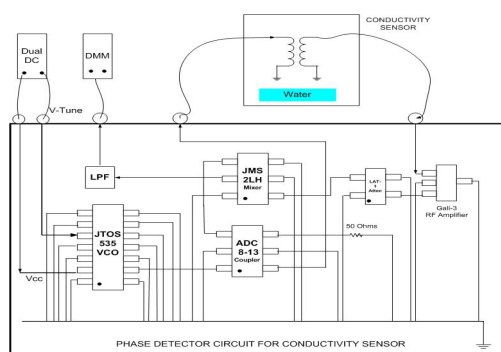


Figure 5. C sensor based on phase measurement.

The electrode system is also being constructed at this time. It is a copper-platinum-black platinum system over Cu/LCP. The pressure-sensing mode for the CTD is based on the material deformation of the copper clad LCP. Two distinct sensors were developed using this approach, relying either on the stretching of the copper or the compression of the LCP under higher pressure environment. Currently the film is being tested for response.

Integrated microchip Ionsprayer. We have focused on the microchip ion sprayer towards modeling and understanding a direct introduction liquid sprayer with integrated ion lensing. Next steps are the fabrication of the integrated chip using our developed LCPMEMS fabrication process for fluidics,

sprayer and integrated lens system. We have implemented an ion lens array testbed and have achieved 25% ion transport, compared to the industry standard of 1-3 %.

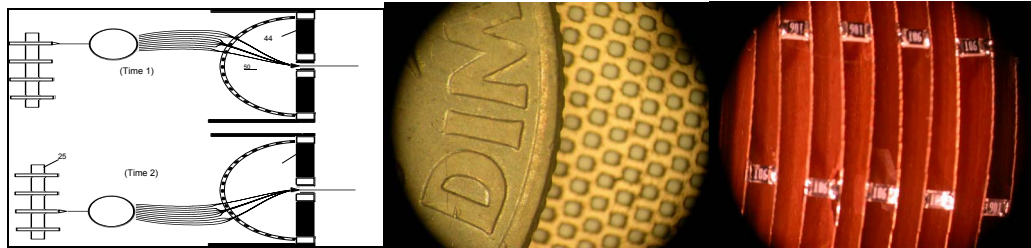


Figure 6. SIMION modeling of a spray chip (at left). LCP/Cu mesh (center) and voltage ladder lens stack (right).

Sensor Array Packaging. Also included in the project is the creation of a system in a package approach towards integrated marine microsensors. We have proceeded with a polyimide folded flex system in a package approach. We have further involved NAVSEA –Crane in the PCB board design who is supporting the design and fab effort.

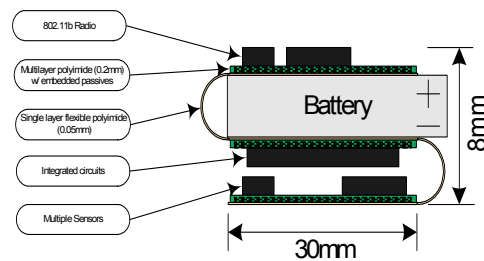


Figure 7. Folded flex system in package approach for the wireless microdata logger portion of the integrated fieldable microsystem.

RESULTS

We have contributed to advances in the field of Maritime MEMS and the techniques needed to fabricate microfluidic-based systems for field analytical purposes. We have initiated primary work in the development of PCBMEMS, an emerging field that will result solely from this funded project. Our current focus is to push integration of the explosive lab prototypes into the field for homeland security support. This proof of technology demonstration has impact for AOSN, Ocean Observing Systems, Navy Sensor Grids and Operational Oceanography and NAVSEA will benefit from the new manufacturing and technology approach and additionally the creation of a microdata logger.

PATENTS, PUBLICATIONS, TECHNOLOGY TRANSFER

Fries, D.P., Securing Harbor Systems, SeaMIST: Sea Micro Integrated Systems Technology, (Invited) COMMS-Comm. Microsystems Symposium 2002, Sept. 2002, Ypsilanti, MI

Broadbent, H., Fries, D.P Steimle, G., Natarajan, S., Weller, T, PCBMEMS Using Liquid Crystal Polymer, LCP Symposium, (Invited) ARMY –IPC, Huntsville, AL, Oct. 2002

M. L. Janowiak, D. P. Fries, A. M. Cardenas-Valencia, M.L. Hall. Water Field Analysis Using Solid Phase Extraction Analytical Systems on Mobile Robotic Vehicles. (Invited) 5th International Symposium on Advances in Extraction Technologies (ExTech 2003), Tampa, FL March, 2003.

D. Fries, H. Broadbent, G. Steimle, A. Cardenas-Valencia. “Liquid Crystalline Polymer- Based PCBMEMS.” Paper presentation and Proceedings of the 226th ACS National Meeting September 7-12, 2003, New York, NY.

D. Fries, G. Steimle, H. Broadbent, S Ivanov, S. Natarajan, A. Cardenas, M. Janowiak, T.Weller, R. Benson and J. Fu. ” *Maskless Lithographic PCB/Laminate MEMS for Marine Sensing Systems.*” Paper presented at the 35th International Symposium on Microelectronics. International microelectronics and Packaging society. IMAPS 2002 Conference at Denver Co, September 4-6, 2002

H. Broadbent, D. Fries, G. Steimle, S Ivanov, S. Natarajan, T.Weller. “PCB/MEMS Salinity Analyzer.” Poster presented at the conference for New Horizons for Environmental Analytical Chemistry through Minutization and Nanotechnology, 6th Euroconference on Environmental Analytical Chemistry at Peer, Belgium on October 18-22, 2002.

H. Broadbent, G. Steimle, D. Fries, S Ivanov, S. Natarajan, A. Cardenas, M. Janowiak, T.Weller, R. Benson and J. Fu. “Maskless Lithographic LCP PCB/MEMS for Field Sensors.” Paper presented at the Liquid Crystalline Polymer Material Processing and Applications Symposium, PMTEC conference at Huntsville, AL, on October 29, 2002.

D. Fries, A. Farmer, H. Broadbent, G. Steimle, M. Janowiak, S. Natarajan, S. Ivanov. “PCBMEMS (Microelectricalmechanical Systems) for Marine Sensing.” Paper presented at Aquatic Sciences Meeting, ASLO conference at Salt Lake City, UT, February 9-14, 2003.

C. Fries, D. Fries, H. Broadbent, G. Steimle, E. Kaltenbacher, J. Sassarath. “Direct Write Patterning of Microchannels.” Paper presented and published in Proc, of the First International Conference on Microchannels and Minichannels being held at Rochester, NY, USA on April 24-25, 2003.

A. Cardenas-Valencia, D. Fries, G. Steimle, H. Broadbent, L. Langebrake, R. Benson. “Fabrication of micro-actuated galvanic cells as power sources for Lab on chip applications by means of novel PCB/MEMS technology.” Paper presented and published in Proc, of the First International Conference on Microchannels and Minichannels held at Rochester, NY, USA on April 24-25, 2003.

D. Fries, M. H. Broadbent, M. Janowiak, A. Cardenas-Valencia, S. Ivanov, R. Benson, G. Steimle Printed Circuit Board Based Oceanographic Microfluidic Systems. Poster accepted for presentation and paper submitted for the proceedings volume of mTAS 2003, The 7th International Conference on Miniaturized Chemical and BioChemical Analysis Systems, October 5-9, 2003, Squaw Valley, CA.

Cardenas-Valencia, A. M.; Fries, D.; Broadbent H. Lanegbrake, L. and Bensor R. F. Micro-actuated Aluminum Galvanic and semifuel cells for Powering Remote Lab on a Chip Applications Poster accepted for presentation and paper submitted for the proceedings volume of mTAS 2003, The 7th International Conference on Miniaturized Chemical and BioChemical Analysis Systems, October 5-9, 2003, Squaw Valley, California USA.

Fries, D.; Broadbent, H.; Steimle, G.; and Cardenas-Valencia, A. M., Liquid Crystalline Polymer-Based PCBMEMS. Paper presented at the 226th ACS National Mtg Sept 7-12, 2003, New York, NY.

A. Cardenas, V. R Challa, D. Fries, L. Langebrake, R. F. Benson and S. Bhansali. A Microfluidic Galvanic Cell as an on-chip power source. Sensors and Actuators B, Chemical, Vol 95/1-3, 2003, pp 406-413,

A. Cardenas-Valencia, A. M., Broadbent H.; Langebrake, L, Samson S. and Steimle E. Development of an Environmental Corrosiveness Sensor. Paper presented at the 13th Annual U.S. Navy & Industry Corrosion Technology Exchange Conference, held on July 14-17, 2003 at the Hurstbourne Hotel & Conference Center in Louisville, Kentucky.

Cardenas-Valencia, A. M.; Fries, D. P. Steimle, G; Broadbent, H.; Langebrake, L. C.; Benson R. F. Fabrication of micro-actuated galvanic cells as power sources for Lab on chip applications by means of novel PCB/MEMS technology. Paper presented and published in Proc. of the First International Conference on Fuel Cells being held at Rochester, NY, USA on April 24-25, 2003.

Cardenas-Valencia, A. M.; Fries, D. P.; Langebrake, L. C.; Benson R. F. Rapid design, fabrication and optimization of a single shot thermo-pneumatic micro-actuation for the delivery of minute amounts of liquids. Paper presented and published in Proc. Of the the First International Conference on Microchannels and Minichannels being held at Rochester, NY, USA on April 21-24, 2003.

A. Cardenas, V. R Challa, D. Fries, L. Langebrake, R. F. Benson and S. Bhansali. A Microfluidic Galvanic Cell as an on-chip power source. Paper presented and published in Proc. Of The 16th European Conference on Solid-State Transducers September 15-18, 2002, Prague, Czech Republic

Xiaojuan Fu, Robert F.Benson, J. Wang and D. Fries, "Remote Underwater Electrochemical Sensing System for Detecting Explosive Residues in the Field", Sensors and Actuators, B, July 2003, in press.

David Fries, Xiaojuan Fu, and Eric Steimle, "A Portable Mobile Underwater Electrochemical Sensing System for Detecting Explosives in the Field ", Sea Technology, September 2003, submitted.

X.J.Fu and D.Fries, "A Novel Fabrication of a MEMS Nitro group Electrochemical Sensor by a Maskless Lithographic PCB/Laminate Technique", Electrochemistry Communications, in preparation.

X.J.Fu and D.Fries , "A Novel PCBMEMS Planar Electrochemical Sensor for Detecting Explosive Residues in the Field", Electroanalysis, in preparation

M. L. Janowiak, A. M. Cardenas-Valencia, M. L. Hall, and D. P. Fries Solid-Phase Extraction - Reflection Spectroscopy Fieldable Analytical System for Water Analysis,. In preparation.

T.Y. Kim, K. Alhooshani, A. Kabir, D.Fries, A. Malik, Sol-Gel Titania Hybrid Organic-Inorganic Coating for On-Line Capillary Microextraction/HPLC, Analytical Chemistry, submitted

J. Wang, G. Chen, Microchip Capillary electrophoresis with electrochemical detector for fast measurements of aromatic amino acids, Talanta, 60 (2003) 1239-1244

PATENTS PENDING

D. Fries, G. Steimle, H. Broadbent. Self-propelled Sensor Apparatus for In-Situ analysis of Environmental Parameters. Patent Application No. 10/319,683. December 13, 2002.

A. Cardenas, L. Langebrake, D. Fries and R. Benson Actuated Electrochemical Power Source. Invention disclosure submitted to Division of Patents and Licensing USF. December 9th 2002.

David P. Fries and Michelle L. Janowiak, Sensor Apparatus for In Situ Analysis of Chemical and Biological Species Patent filed Nov. 25, 2002.

A, Malik, W.Li, D Fries, Method for Sample Preconcentration in Capillary Electrophoresis using Sol-Gel Media, Patent Submitted